

SEMI C51-0706

SPECIFICATIONS AND GUIDELINES FOR XYLENES

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NOTICE: This document was completely rewritten in 2006.

NOTICE: Paragraphs entitled NOTE are not an official part of this document and are not intended to modify or supercede it.

1 Purpose

1.1 The purpose of this document is to standardize requirements for xylenes used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results.

2 Scope

2.1 The scope of this document is all grades of xylenes used in the semiconductor industry.

2.2 This material is a mixture of ortho, meta, and para isomers and ethyl benzene. Xylene (o-, m-, and p- isomers and ethyl benzene) 99.0% min and Ethyl benzene 18% max.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 None.

4 Referenced Standards and Documents

4.1 SEMI Standards

SEMI C1 — Guide for the Analysis of Liquid Chemicals

4.2 ASTM Standards¹

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 None.

6 Physical Properties (for information only)

6.1 Density at 25°C 0.86 g/ml

6.2 Boiling Range 136–144°C

7 Requirements

7.1 The requirements for xylenes for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

8.1 Assay — Analyze the sample by gas chromatography (see SEMI C1, ¶ 7.2 ‘Assay by Wide Bore Column Gas Chromatography’). The parameters cited have given satisfactory results.

- Column: 30 meter × 530 micron I.D. fused silica capillary, coated with 5 micron film of DB-Wax or equivalent (100% methyl silicone which has been surface bonded and cross linked).

¹ American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555, www.astm.org.

- Column Temperature: 40°C isothermal for 5 minutes, then programmed to 200°C at 10°C/min.
- Injector Temperature: 175°C; Detector: Thermal Conductivity or Flame Ionization, Detector Temperature: 250°C.
- Sample size: 0.2 µL splitless; Carrier Gas: Helium at 3 mL/min:
- Approximate Retention Times (min): Toluene – 14.4, Ethyl benzene – 17.0, p-xylene – 16.9, m-xylene – 17.2, o-xylene – 17.8.

8.2 *Color* — Dilute 2.0 mL of platinum-cobalt stock solution (APHA No. 500) to 100 mL with water. Compare this standard (APHA No. 10) with 100 mL of sample in Nessler tubes. View vertically over a white background. The sample must be no darker than the standard.

8.3 *Acidity* — To 25 mL of water in a glass-stoppered flask, add 10 mL of sample and 0.1 mL of phenolphthalein indicator solution. Add 0.01N sodium hydroxide until a slight pink color persists after shaking for one-half minute. Add 38 mL (33 g) of the sample, mix well, and titrate with 0.01N sodium hydroxide until the pink color is reproduced. Not more than 1.0 mL of the sodium hydroxide solution should be required.

8.4 *Residue after Evaporation* — Evaporate 232 mL (200 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1, ¶ 4.15 ‘Residue after Evaporation’).

8.5 *Water* — Add 25 mL of methanol to a dry titration flask and add Karl Fischer (KF) reagent to a visually or electrometrically determined endpoint that persists for 30 seconds. Add 23 mL (20 g) of sample, taking care to protect the sample and contents of the flask from moisture. Stir vigorously and titrate with Karl Fischer reagent to the same endpoint.

$$\% \text{ Water (H}_2\text{O)} = \frac{\text{mL KF reagent} \times \text{KF factor (g H}_2\text{O/mL)} \times 100}{\text{Weight of sample (g)}}$$

8.6 *Chloride* — To a 100 mL separatory funnel, add 12 mL (10 g) of sample and 40 mL of water. Shake well for 30 seconds and allow the two layers to separate. Discard the sample (upper) layer. To a 20 mL portion of the water layer add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution. Any turbidity produced should be no greater than that of a standard containing 0.015 mg of chloride ion (Cl) in an equal volume of solution and the amounts of reagents used.

8.7 *Phosphate* — To 12 mL (10 g) of sample, add 10 mL of sodium carbonate reagent solution and evaporate to dryness on a steam bath in a hood. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand at room temperature for 2 hours. Any blue color produced should be no greater than that produced when 0.01 mg of phosphate ion (PO₄) is treated like the sample.

8.8 *Arsenic and Antimony (as As)* — Evaporate 116 mL (100 g) of sample in a 250 mL beaker to a small volume in a hood. Add 50 mL of water and again evaporate to a small volume. Repeat the evaporation three times with water additions. Do not allow to go to dryness. Add 5 mL of nitric acid and 5 mL of sulfuric acid and evaporate to dense fumes of sulfur trioxide. Cool, cautiously add 10 mL of water, and again evaporate to dense fumes of sulfur trioxide. Cool, and cautiously wash into a generator flask with water to make a volume of 35 mL. Proceed as described in SEMI C1, § 7.6 ‘Trace Arsenic (and Antimony) Determination’, starting with the sentence that begins, “Swirl the flask...”. Any red color in the silver diethyldithiocarbamate solution of the sample should be no greater than that of the standard containing 0.001 mg of arsenic (As).

8.9 *Trace Metal Analysis*

8.9.1 *Boron* — To 116 mL (100 g) of sample, add 0.10 mL of 10% sodium hydroxide, and evaporate to dryness in a nitrogen atmosphere using a covered chamber such as a Thiers assembly or equivalent. Dissolve the residue with 2 mL of water and 0.10 mL of hydrochloric acid. Transfer to a test tube, cool in an ice bath, and add 10 mL of sulfuric acid. Add 10 mL of carminic acid solution (¶ 8.9.1.1) and remove from the ice bath. Allow to stand for 45 minutes. Prepare a standard containing 0.01 mg of boron treated exactly as the sample (¶ 8.9.1.2). Run a complete blank determination on 2 mL of water. Measure the absorbances of the sample and standard against the blank at 585 nm using 5 cm cells. The absorbance of the sample solution should be no greater than that of the standard.

8.9.1.1 *Carminic Acid Solution* — Dissolve 0.05 g of carminic acid in 100 mL of sulfuric acid and shake until dissolution is complete.